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The Metallurgist.

Supplement to The Engineer, *December, 1935.*

Research on Pressed Steel.

A YEAR or so ago a number of manufacturers interested in sheet metal suggested that the Institution of Automobile Engineers should undertake an investigation on various problems related to deep-drawing and pressing of steel. The matter was referred to the Research and Standardisation Committee of the Institution, the function of which is to carry out co-operative research on behalf of the motor industry. Its affiliated members embrace industries producing all kinds of materials and components for motor vehicles, and, in this connection, it is common knowledge that sheet metal is used extensively, *e.g.*, for body panels, chassis frames, wheels, &c. One outcome of this was the discussion on cold pressing and drawing which took place last March, and afforded a valuable review of the problems involved and of the directions in which researches should be guided in an attempt to solve them. Another was the formation of a sub-committee to consider future developments. This Sub-committee has held several meetings at which different aspects of the subject have been discussed.

Thus it was suggested that what was required was the preparation of specifications by users and sheet makers to meet different categories of requirements, such specifications covering chemical composition, processing of material, grain size, structure, physical characteristics, &c., and, if the material was required for deep drawing, a drawing test on a standard pressing should be part of the specification. It was pointed out that manufacturers are still very much in the dark as to whether any particular consignment of steel will give satisfaction in the press shop, and that a simple shop test for pressing quality was required which would enable a faulty batch of steel to be discovered.

On the other hand, some members of the Sub-committee considered that for the time being no simple test could be established which would permit the elimination, with certainty, of a faulty batch of steel. The difficulty, in this connection, is that the materials which respond similarly to certain tests may not be uniform in their behaviour on cold pressing.

From these discussions, which showed the demand for both tests and further knowledge, there emerged the assertion that fundamental research was required, and should constitute the first step. It was pointed out that a programme of empirical comparative testing would be wasteful in the present state of our knowledge, and that what was required was a research into the fundamental properties of materials which determine pressing quality. Another proposal for the research was that "it should be directed towards reaching an understanding of the phenomena which arise when a material is subjected to deep-drawing," while the ultimate object should be "concerned with the development of a better practice in the deep drawing of known materials."

It was suggested that the property which is of the greatest importance in deep-drawing operations is that of plasticity, which is usually measured in terms of percentage elongation and reduction of area, and that an investigation of the factors governing their relationship would be useful. Again, it appears that a low resistance to deformation by compression, together with a high tensile strength, would be an ideal combination for deep-drawing operations, and the factors governing the development of these properties and their effect on drawing properties could be studied.

A proposal for the use of a standard form of drawn specimen, or range of pressings, involving the experimental adaptation of a press fitted with indicating or recording instruments received the immediate support of the Sub-committee, which has since concentrated on developing the idea.

The behaviour of the steel in the press could be correlated with mechanical and physical tests and the influence of the following factors, amongst others, could be studied :—Grain size, lubrication, speed of deformation, chemical constituents to suppress ageing, drawing temperature, overstrain in tension, and compression, &c. It was emphasised that, as regards material, the study should begin at the ingot stage, and that the fullest co-operation of the supplier was essential. In addition, the investigation should include a study of the modification of structure in the drawn specimens. Attention was also called to the need for a concurrent mathematical analysis of the stresses and strains involved.

At this stage the co-operation of Principal Edwards (Swansea) and Professor Andrew (Sheffield) was invited, and a provisional programme of research was drawn up, involving work at both the above-mentioned universities. Briefly, it is proposed, at Swansea, to make a careful study of the drawing properties of mild steel and other sheet metals, while the problems associated with strip are to be dealt with at Sheffield.

The provisional programme has been discussed at a conference of representatives of manufacturers and other interested bodies. This conference proved to be most useful in bringing to bear on the proposals constructive criticism and valuable advice. The importance of co-operating with the Ingots Committee of the Iron and Steel Institute was emphasised, and an assurance of the sympathetic interest of the British Iron and Steel Federation was received. Among other important matters mentioned in the discussion were the need for including some work on non-ferrous metals and alloys as well as steel, and the importance of investigating such properties as strain age embrittlement in connection with the behaviour of deep

drawing steels. The proposal for an experimental press received unanimous support; in fact, the desirability of having two experimental presses, one at Swansea for sheet metal and a smaller one at Sheffield for strip metal, was mentioned. A considerable amount of time was devoted to a discussion of the best types of press and design of dies for these experimental purposes.

It is obvious that a programme of the type under discussion will need considerable financial support, and it was largely with the object of securing such support and of widening the interest in the subject that the conference was called. The tone of the meeting made it clear that the scheme was likely to be successfully launched. Mr. C. G. Williams, the Director of Research for the Institution of Automobile Engineers, was able to announce that six firms had made definite promises to subscribe, while several others had the matter under sympathetic consideration, but had not decided on the amount of their contribution. Nevertheless, much remains to be done in the direction of securing adequate financial support. The programme, though it will be closely checked at all points to bring it into line with practical requirements, is bound to be a very large one. It is most desirable that there should be no delay in putting it into effect, and those who have contributed to the successful inauguration of the research deserve the wide and generous support of industry in seeing it through.

The Recent International Congress.

THE Congrès International des Mines, de la Métallurgie et de la Géologie Appliquée is a quinquennial event, and was held this year in Paris during October, when over one thousand technical and scientific workers attended, including many notable metallurgists. The technical institutions of twenty-six countries were represented, and while the official contingent from this country was small (only just exceeding that of Uruguay), several British metallurgists, including Sir Robert Hadfield and Dr. H. Moore, took a prominent part in the proceedings. Perhaps the main impression left by the Congress was that of supremely good organisation, and Monsieur Léon Guillet, the President of the General Organising Committee, is to be highly congratulated on the successful outcome of the task which his Committee undertook so efficiently and artistically. The magnificence of the opening session at the Sorbonne, at which the President of the Republic was present, was most impressive, and set a standard which was maintained throughout the whole of the functions of the Congress. Of the works visited, those of the Renault Company were particularly instructive and varied in interest, showing really up-to-date plant and methods of assembly, and illustrating the tendency towards co-ordinating the whole range of manufacture from the raw materials to the finished product. The laboratories are exceptional in design. Another pleasure was the inspection at the School of Mines of the large and well-arranged geological collection. Of the great number of papers presented, covering a wide range of subjects, it is difficult to make any selection for special mention. A complete session was devoted to the discussion of "flake" defects in steel, an old but still absorbing topic. Among the contributions relating to recent advances, those on new products, such as refined aluminium and beryllium, on the nitriding of magnesium and aluminium, on light alloys and on testing methods, were of particular interest. The Congress afforded a good illustration of the value of scientific co-operation, and the publication of the full proceedings should serve to correlate the information available on a variety of metallurgical topics of current interest.

The *Scaling* of Iron and *Steel*.

THE value and usefulness of materials to be used at elevated temperatures is largely dependent on their resistance to oxidation or scaling.

Apart from this, any scaling which takes place during the manufacture of iron and steel articles, with the exception of a few cases to be mentioned later, may cause a waste of material and even impair the quality of the finished product.

This waste arises from the conversion of an important percentage of the metal into oxide during the reheating and rolling or forging of the material into bars, sheet, and wire. The quality of the material may be impaired by the oxidation leading to surface defects. Such defects are particularly liable to occur if the steel is not "free scaling," in which case some scale adheres to and becomes embedded in the surface when the steel is subsequently rolled or drawn. In addition, the rolls and dies themselves may be damaged by such adhering scale.

Another aspect of the ill effects of scaling may be seen in the heat treatment of steels. The scale formed on heating may thermally insulate the steel during the quenching operation and prevent it cooling sufficiently quickly to harden satisfactorily, whilst in some cases the scale may partially flake off, causing non-uniform hardening. Where scaling of a carbon steel occurs, it is generally accompanied by decarburisation of the material in the vicinity of the surface. Surface removal of the carbon by reaction with the scale tends to be equalised by the diffusion of carbon from the interior to the surface, but the latter effect is never sufficiently rapid to make up the loss. The three phenomena, oxidation of the steel, its decarburisation, and the diffusion of the carbon, are all accelerated with increase in temperature, but not to the same extent. In addition, they are affected by the carbon content of the steel and the presence of any alloying elements. The importance of the phenomena lies in their effects on the hardening qualities of carbon steel articles. Such articles may exhibit a soft cutting edge or increased liability to surface cracks resulting in fatigue failure.

Even when the scale has been removed, the effects of oxidation may still persist in the underlying steel surface, in that globules and spots are formed therein during oxidation.[1]

As previously mentioned, limited scaling is sometimes intentionally sought, instances of which are the thin adherent coatings on blued steel or the Bower-Barf coating, or that on planished Russian iron now seldom used. In some welding operations also the presence of scale is considered an advantage.

MECHANISM OF SCALING.

Contributions to a truer understanding of the mechanism of oxidation have been made by a number of workers during the past few years. U. R. Evans[2] has isolated and examined the thin surface films formed during the oxidation of iron on exposure to

air at ordinary temperatures, and found them to be similar in appearance and composition to the thicker films obtained from iron which has been heated sufficiently to give interference tints. A study of these films has enabled a number of conclusions to be drawn as to the mechanism of scaling.

Pilling and Bedworth[3] determined the rates of oxidation of a number of metals under varying conditions, and deduced therefrom a number of principles. They showed that it is possible to assign the majority of metals to two main classes according to the relations existing between the densities of the metals and the oxide. The first class contains metals whose oxides occupy a volume less than that of the metals from which they are produced, and includes the ligher metals with the exception of aluminium. The oxide layer produced is necessarily porous, and will not seriously obstruct the passage of oxygen to the metal below. The thickness of the film x produced at constant temperature is proportional to the time of heating t. $x=k\,t$. Metals of this class cannot be used for practical service at elevated temperatures, because they " burn in air."

The metals of the second class, which includes iron and steel, occupy a volume smaller than the uncompressed volume of the oxide. The oxide produced on the surface of the metal on heating in air is nonporous, and in a state of lateral compression. This coating tends to diminish the rate of formation of a thicker coating.

Pilling and Bedworth recognised the action of oxygen in dissolving in the scale and diffusing through to the metallic core, but a great advance was made when Pfeil[4] established by his work on the oxidation of iron that excess iron diffused outwards through the oxide scale. Other workers in this field have found evidence in support of this hypothesis, although in some cases some modification of it has been found necessary.

It can easily be understood that under normal oxidising conditions of iron and steel, countercurrent diffusion of oxygen inwards and iron outwards must occur. If oxygen only diffused through the scale to the iron, forming more scale beneath that already formed, the first-formed scale would be forced outwards as the result of the expansion. Being nonplastic at anything but very elevated temperatures, the scale would be cracked by the expansion, but this, in fact, does not happen. On the other hand, if scaling proceeded only by the solution of iron in the scale, the passage of iron outwards to the surface would result in the continuity between the iron and scale being lost, in which case scaling would cease.

Under normal conditions, the rate of passage of oxygen inwards through a laterally compressed oxide on a metal (and likewise the rate of diffusion of metal outwards) will be inversely proportional to the thickness of the oxide, and the velocity of oxidation can be expressed by a simple parabolic equation $W^2=K\,t$, where W is the amount of oxygen absorbed, t is time, and K a constant.

This law has received ample experimental verification[5], but several authorities do not regard it as very rigid.

It may be mentioned here that in the case of some non-ferrous alloys a change in state of the oxide film due to sintering or annealing may cause deviation from the parabolic law.[6] The progress of the process may be studied by an analysis of the oxidation curve.

In the absence, therefore, of complicating factors, the rate of oxidation of metals which oxidise according to the parabolic law would decrease with time, rendering this class of metals suitable for use at moderately high temperatures. The parabolic law is obeyed by physically perfect oxides, but under practical conditions strict adherence to the law is not always found in the case of iron and steel, owing to disturbing physical influences.[7] The matter appears complicated when iron and steel are oxidised in air, because of the existence of three separable oxide layers on the metal. Nevertheless, the recent work of Portevin, Prétet, and Jolivet has shown that the parabolic law is accurately obeyed when electrolytic iron is oxidised between 825 deg. and 1000 deg. Cent. In examining the three layers of scale on iron, Pfeil[4] observed that the boundary between the inner and middle layers corresponded in position and appearance to the original surface of the specimen. The oxygen content of the scale increased on passing from the surface of the metal core to the surface of the scale, whilst the iron content correspondingly decreased. Up to four phases may be present in the scale, the outer surface of which commonly consists of a thin layer of ferric oxide, the intermediate thicker layer of magnetite, while the bulk of the scale deposit is made up of the iron-rich oxide, the so-called " ferrous oxide " phase, or Wüstite. The above formation is not rigid, for the lowest phase never corresponds to the exact composition FeO, and a range of compositions is possible in the other layers. Furthermore, the ferrous phase decomposes on cooling, giving a eutectoid structure consisting of particles of magnetite in a ground mass of the ferrous phase.

When oxidation takes place in an atmosphere deficient in oxygen, scale deposits may be obtained in which only the Wüstite phase appears, and the surface of the scale consists of idiomorphic crystals.[4]

The atmosphere also has a bearing on the thickness of the ferric oxide layer which may be thinner in that produced in steam-air mixtures than in that produced in air alone.[1] In the case of scale produced at a temperature below 570 deg. Cent. the Wüstite scale should be absent, since this phase is believed to be unstable below 570 deg. Cent., and the layer adjacent to the steel should consist of magnetite or ferric oxide.

The existence of the ferrous phase is of industrial importance in the de-scaling of steel by means of acid. The experiments of Winterbottom and Read[8] show that ferrous oxide is much more readily soluble than the other oxides, and low temperature scale from which the ferrous oxide is absent is much more difficult to remove by pickling.

It will be realised that the structure of scale on iron is very complicated, and especially when other elements are present. A complete equilibrium diagram of the iron-oxygen system would do much towards a better understanding of the phenomena of oxidation of iron and steel. Much valuable work in this direction has already been done, particularly by Benedicks and Löfquist,[9] Schenck and Dingmann,[10] Pfeil,[11] Mathewson, Spire, and Milligan,[12] Schenck and Hengler,[13] and Jette and Foote[14]. The results obtained by the various workers, however, do not agree at all closely, particularly in the section of the diagram covering the range 74 to 76 per cent. of iron, differences of about 300 deg. Cent. being given for melting points in this region. The existence of the compound FeO is also doubted, it being suggested that the oxide phase richest in iron is a solid solution extending over the range of approximately 75 to 77 per cent. iron and termed Wüstite. The lack of agreement regarding the diagram may be attributed very largely to the many experimental difficulties

which must be overcome if accurate results are to be obtained.

SCALING OF ALLOY STEELS.

Although a great deal of experimental work has been done on the scale deposits on alloys of iron, the subject is only in its infancy. It appears that small additions of alloying elements do not result in any fundamental change in the mechanism of oxidation, but the constitutions of the scale deposits produced are materially affected. In the case of chromium steel, Pfeil[14] found that the chromium is concentrated in the inner scale layer, and Portevin and his collaborators[15] demonstrated the existence of an external layer of Fe_2O_3, an intermediate layer of Fe_3O_4 loaded and modified by chromium oxide. Rickett and Wood[16] who also studied the oxidation of iron-chromium alloys in both oxygen and hydrogen sulphide atmospheres, considered that the mechanism of oxidation could be explained on the countercurrent diffusion hypothesis.

The nature of the scale on nickel steel [17] may be regarded as evidence strongly in favour of the countercurrent mechanism of oxidation. Oxidation of the iron only takes place and nickel is to be found in the scale in the form of small metallic globules, which consist of nickel-rich nickel-iron alloy in equilibrium with the Wüstite ground mass. The outer portion of the scale is completely free from nickel, while in the innermost layer the nickel to iron ratio is two to three times as great as in the original steel.

Iron-aluminium alloys have received some attention, but comparatively little is known with regard to the constitution of the scale formed on them. G. Sykes and J. W. Bamfylde[18] have recognised two types of scale. When oxidation is slow a fine powder is formed, varying in colour from red to white with increasing temperature and aluminium content; rapid oxidation causes the formation of a dense, black adherent scale. The white powder consists of aluminium oxide. Silicon[15] in itself does not appear to retard the rate of oxidation of iron, but assists in increasing the resistance of alloy steels.

Copper bearing steels have been shown by F. Nehl[19] and W. Radeker[20] to produce a scale having a copper-rich metallic phase at the steel surface. Radeker found that a cobalt steel behaved in an exceptional manner in that the inner scale layer contained less cobalt than the outer layers. The results obtained with a steel containing vanadium were also remarkable in that the vanadium concentration was at a minimum in the middle scale layer.

Manganese does not appear to tend to become concentrated in any scale layer, but is distributed fairly uniformly throughout.

The results of various workers on the oxidation of alloy steels appear to show that the process of diffusion of various elements in the scale is the chief factor governing the constitution and incidentally the amount of scale formed. The relative rates of diffusion of iron and the alloying element will govern the distribution of the alloying element in the scale. It has been found that the greater the amount of alloying element added to develop heat-resisting properties the greater, within limits, is the effect in decreasing the tendency to scale. Pfeil suggests that the rate of diffusion of oxygen or of iron in the scale deposit is gradually lessened, owing to the blocking of the diffusion path. This may result merely from the presence of the alloying atoms in the inner layer or Wüstite phase, but may also be due to the gradual replacement of the Wüstite phase by another phase through which iron and oxygen diffuse very much less readily.

SCALING IN COMPLEX GASES.

In industrial practice the conditions of scaling are naturally complex. Apart from temperature variations, there are usually present in furnace atmospheres a number of highly corrosive gases at high temperatures. The rate of attack may be expected to be much greater than in air. Heat-resisting steels are necessary to withstand the action of these gases, and it is extremely difficult to simulate industrial conditions in the laboratory in order to assess the individual or combined effects of the gases on steels. Fundamental studies are required and work is proceeding in this direction, but the multiplicity and complexity of the problems will naturally delay for some considerable time a complete understanding of the various factors.

The velocity with which the stream of oxidising gases passes over the surface of the steel appears to have an influence on the rate of scaling.[21] The rate of oxidation rapidly increases with the velocity up to a certain critical figure, after which the rate becomes constant. This is due to the fact that the impoverishment of the oxidising atmosphere by reducing or inert gases occurs at the lower velocities, whilst with more rapid velocities these gases are swept away. At the same time the supply of oxygen is augmented to a value sufficient for normal oxidation. The import of this phenomenon is that the furnace design in itself has a direct influence on the amount of scaling that can take place, other conditions being equal.

Furnace atmospheres may be made up of combinations of gases, such as nitrogen, steam, carbon dioxide, carbon-monoxide, hydrogen, sulphur dioxide, &c. Steam alone is known to produce a scale from which the ferric oxide and magnetic oxide layers are absent, a similar result being produced by carbon dioxide alone. The rate of oxidation in steam, however, is much more rapid than that in air, whilst in carbon dioxide the rate is much slower than in air. This can be explained by the fact that the ferrous phase can accept oxygen much more rapidly from the steam than it could from magnetite, were it present. The carbon monoxide molecule, however, is much less mobile than the hydrogen molecule, both being respectively products of the oxidation. This results in the hydrogen molecule being able to diffuse away comparatively rapidly from the scale surface, whilst carbon monoxide tends to remain at the surface and dilute the reacting gases. Murphy[22] found that in town gas, burnt completely without excess of air, the addition of 0·2 per cent. of sulphur dioxide doubled the scaling rate at temperatures over about 1100 deg. Cent., but that when the gas was burnt with a deficiency of air, the effect of the addition of sulphur dioxide became less and less pronounced as the atmosphere became more reducing. The steel heated in the sulphurous atmosphere was found to have absorbed sulphur to an appreciable extent.

Upthegrove[23] found that sulphur dioxide was more active at 925 deg. Cent. when the atmosphere contained carbon monoxide than when it contained excess oxygen. This difference according to the temperature is probably due to the formation of relatively harmless H_2S when sulphur dioxide is added to a reducing atmosphere at high temperatures, whilst no conversion takes place at lower temperatures.

Upthegrove and Murphy[24] in their experiments on scaling in steam-carbon dioxide atmospheres at a temperature of 925 deg. Cent. showed that the rate

of scaling increased as a linear function over the range 0 to 70 per cent. steam, but between 70 and 100 per cent. steam the scaling remained almost constant.

The above brief outline of the research work carried out on the scaling of iron and steel will give some indication of the great need for much further work in some directions.

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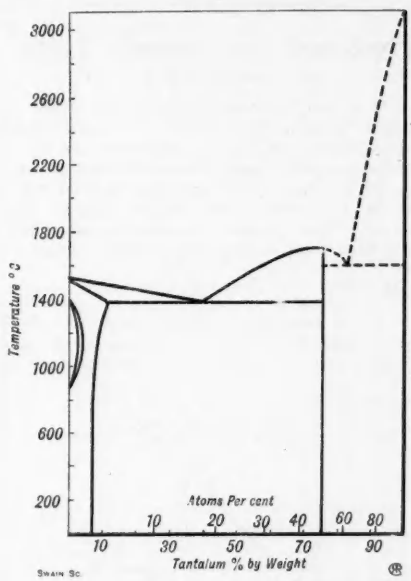
The Iron Tantalum Diagram.

IN view of the fact that it is a long time since tantalum steels were first investigated, that there is now a considerable output of ferro-tantalum, and that the addition of tantalum to nickel-chromium austenitic steels is being advocated, it is surprising that so little is known of the constitution of the iron-tantalum alloys. Iron is generally supposed to form a compound with tantalum, and F. Wever* classes tantalum among the elements which form a closed γ loop when alloyed with iron. A preliminary survey of the system has recently been made by W. Jellinghaus.† Six alloys, containing 6, 10, 15, 25, 50, and 75 per cent. of tantalum, were made from tantalum metal (obtained from De Haën, Seelze, near Hannover) and carbonyl iron by first sintering, and then melting *in vacuo* in alumina crucibles. The provisional diagram shown below, in so far as it refers to alloys containing up to 50 atoms per cent. of tantalum (76·4 per cent. by weight), is based on a study of the micro-structure, the magnetic properties, and the fine structure of these six alloys. The limit of the γ loop is placed at 4·25 per cent. on the authority of Wever.

The 6 per cent. tantalum alloy solidified to form a homogeneous solid solution, but in the large grains a precipitate in the form of needles was found after quenching from 1200 deg. Cent. in oil. In the 10 per cent. alloy a fine-grained network surrounded the primary crystals in the interior of which fine needles were precipitated, as in the 6 per cent. alloy. The appearance of the alloy with 15 per cent. of tantalum was similar. The maximum amount of tantalum soluble in α iron at the eutectic temperature was estimated at between 6 and 10 per cent.

The occurrence of precipitation shows that with falling temperature the solubility diminishes, but no accurate determination of the position of the solubility line was made. The 25 per cent. alloy showed dendrites of iron-rich solid solution embedded in a



Provisional Equilibrium Diagram of the Iron-Tantalum Alloys.

lamellar eutectic. At 50 per cent. of tantalum, a similar eutectic occurred with a new form of primary crystals which showed rectilinear boundaries with sharp corners, and were only with difficulty attacked by etching reagents. The tantalum content of the eutectic accordingly appears to lie between 25 and 50 per cent. The alloy with 75 per cent. of tantalum is very brittle, and crumbles easily.

The microstructural study was supplemented by an investigation of the fine structure of the alloys. In the Debye-Scherrer spectrograms new lines were observed beside the iron lines when the tantalum reached 15 per cent. and the strength of the lines increased with increasing tantalum content until with 75 per cent. of tantalum (48 atoms per cent.), the iron lines disappeared. This alloy had a hexagonal crystal lattice $a=4·81$ Å and $c/a=1·64$. The calculated size of the unit cell, together with the experimentally determined density 9·88, gave 946 as the total weight of atoms included in the unit cell. Taking into account the effect of the lower tantalum content,

* Mitt K.W. Inst. Eisenforschung, 1931, 13, 184.

† Zeit, anorg. Chemie, Aug. 9th, 1935, Vol. 223, p. 362.

this is in very good agreement with the four-fold formula FeTa (949·6) for the alloy containing 50 atoms per cent. of tantalum. The unit cell of this compound thus contains 4 iron and 4 tantalum atoms. Measurement of the magnetic properties of alloys containing from 15 to 75 per cent. of tantalum shows a gradual fall in B.H. value approaching to zero at about 50 atoms per cent. of tantalum, a further confirmation of the existence of the compound.

The amount of material available was insufficient for thermal analysis, and the only thermal data obtained were the eutectic temperature, 1400 deg. Cent., and the melting point of the compound, 1700 deg. Cent. This being so, the diagram must not be taken for more than the author claims it to be, namely, a first attempt at the outline of a provisional diagram which is consistent with all the observations which he has so far been able to make.

Notched Bar Impact Tests.

By G. BURNS, M. Met.

As it is now well established that there is no calculable relationship between the results obtained in the notched bar impact test with test pieces of different sizes and forms, the equivalence of different types of test piece can only be decided by large numbers of comparative tests on many types of material. The present communication gives the results of several such series of tests carried out in

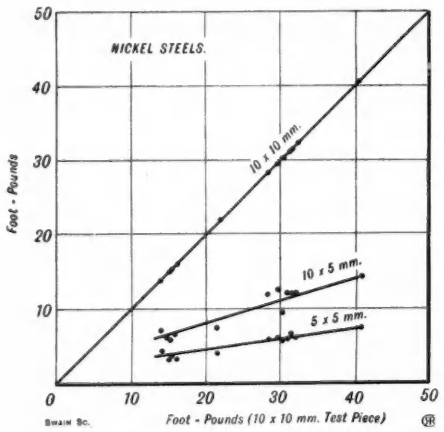


FIG. 1

the Research Department, Woolwich, on a limited range of steels. It is hoped that they may serve to amplify the results of similar comparative tests which may be available elsewhere.

THE B.S.I. SUB-STANDARD TEST PIECES.

The B.S.I. standard notched bar test piece has a cross section of 10 mm. by 10 mm., but to provide for occasions when this size of specimen cannot be cut from the available material, there are two subsidiary standard test pieces 10 mm. by 5 mm. and 5 mm. by 5 mm. in section (B.S. Specification No. 131).

The use of these test pieces in the Izod machine involves an alteration in the height of the grips to give reduced distance between the notch and point of impact. The dimensions of the three test pieces are shown below :

Cross section of test piece.	Depth of notch.	Total height above the notch.	Height of point of impact above notch.
Mm.	Mm.	Mm.	Mm.
10×10	2·0	28	22
10×5	1·5	14	11
5×5	1·5	14	11

The notch in all cases is the 45 deg. V notch, with a root radius of 0·25 mm.

The impact values given by the standard and subsidiary standard test pieces have been compared by the use of material from a number of forgings of nickel and nickel-chromium-molybdenum steels all in the oil-hardened and tempered condition. A three-notch test piece of each size was cut from each steel and tested in the 120 foot-pounds Izod machine, the average values being plotted against the average value obtained with the standard test piece in Figs. 1 and 2. The results obtained with

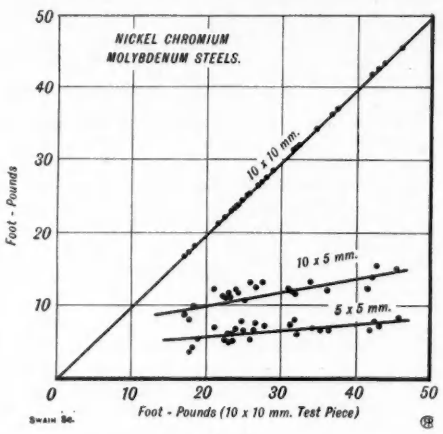


FIG. 2

the smaller test pieces on any one steel were just as consistent among themselves as those obtained with the standard test piece, the mean deviation of individual results from the average for each three-notch test piece being :

For the 10 mm. × 10 mm. test piece	A ..	2·1 foot-pounds
„ „ 10 mm. × 5 mm. „	„ B ..	0·6 foot-pound
„ „ 5 mm. × 5 mm. „	„ C ..	0·3 „

Nevertheless, the ratios of the values given by the smaller test pieces to those given by the standard fluctuated widely, the ratio A : B varying from 1·9 to 3·7, and A : C from 3·6 to 7·5 with a tendency for the higher values to occur more frequently as the impact figure rises. It is thus clear that any attempt to convert the impact figures obtained with the smaller test pieces into the standard impact figure is subject to the possibility of a large error of the order of plus or minus one-third, and as the values given by the smaller test pieces do not rise in the same proportion as those given by the standard test piece, the subsidiary standard test pieces will not discriminate so well between tough and brittle materials as will the British standard test piece.

THE PROPOSED GERMAN SUB-STANDARD TEST PIECE.

As a result of a large amount of work carried out in Germany by Moser and others on the influence of

dimensions of test piece, depth, and sharpness of notch, and other factors affecting the indications and applications of the notched bar impact test, a standard and a supplementary standard test piece have been proposed for adoption in Germany.* The German sub-standard test piece approaches the

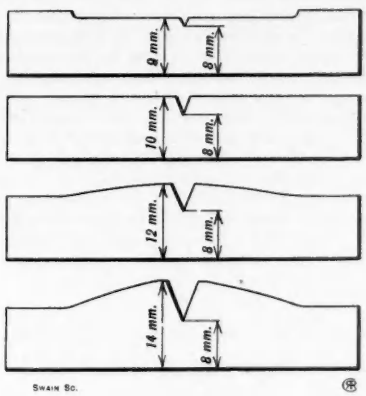


FIG. 3

British standard fairly closely, as the following dimensions show :—

	Section, mm.	Notch.
British standard ..	10 × 10 ..	45 deg. V, 2 mm. deep, 0·25 mm. root radius
German sub-standard	10 × 10 ..	45 deg. V, 3 mm. deep, 0·5 mm. root radius

Before a direct comparison of results obtained with the two forms of test piece is dealt with, the probable effect of these differences may be noted. The effect of increasing the depth of the notch is twofold. Up to a point, it leads to increased concentration of stress at the bottom of the notch, and it also decreases the remaining thickness of metal to be broken. The first factor was investigated by breaking test pieces whose thickness had been increased or reduced in the central portion to accommodate the varying depth of notch while the depth of metal below the notch was unchanged. Tests on five steels and an aluminium bronze showed that

TABLE.—*Effect of Depth of Notch on Impact Value, the Depth of Material Behind the Notch being Constant (8 mm.). Average Impact Value (Foot-pounds).*

Material.	Depth of notch.*	Radius of notch. 0·25 mm.	0·5 mm.	1·0 mm.
0·16 per cent. carbon steel ..	1 mm.	69	88	99
	2 mm.	25	34	70
	4 mm.	17	25	66
	6 mm.	22	29	66
0·55 per cent. carbon steel ..	1 mm.	14	21	29
	2 mm.	9	13	19
	4 mm.	6	10	13
	6 mm.	7	10	14
Ni-Cr steel..	1 mm.	36	41	54
	2 mm.	28	32	43
	4 mm.	26	33	46
	6 mm.	28	32	47
Ni-Cr steel	1 mm.	81	91	97
	2 mm.	64	62	75
	4 mm.	60	67	75
	6 mm.	64	65	74
Ni-Cr steel	1 mm.	76	88	93
	2 mm.	61	69	77
	4 mm.	60	60	72
	6 mm.	61	63	73
Aluminium bronze	1 mm.	44	51	61
	2 mm.	39	41	45
	4 mm.	37	40	45
	6 mm.	37	39	43

* The form of test piece is shown in Fig. 3. Tests were made in the 30 m.kg. Charpy machine.

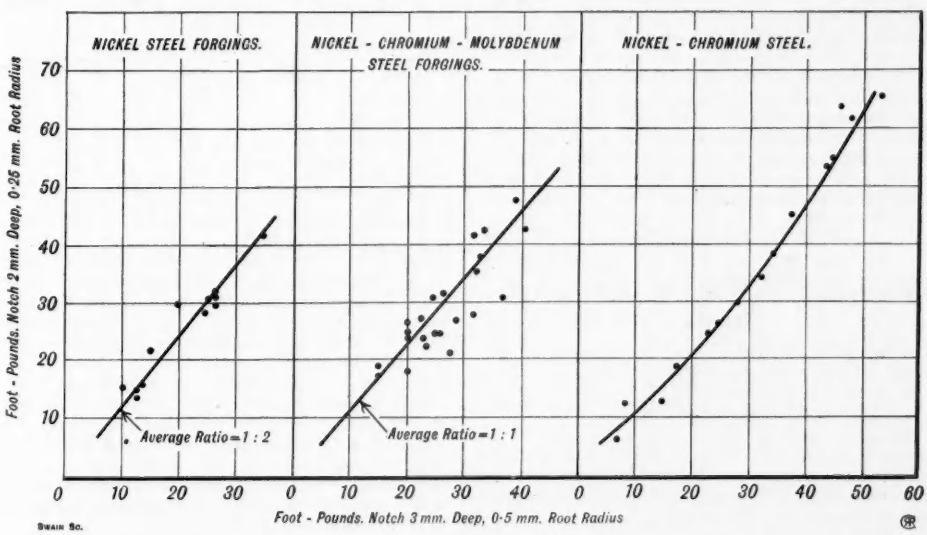

NICKEL STEEL FORGINGS.
NICKEL - CHROMIUM - MOLYBDENUM STEEL FORGINGS.
NICKEL - CHROMIUM STEEL.
Foot - Pounds. Notch 2 mm. Deep, 0·25 mm. Root Radius
Average Ratio = 1 : 2
Average Ratio = 1 : 1
Foot - Pounds. Notch 3 mm. Deep, 0·5 mm. Root Radius
SWAIN SC.

FIG. 4

* Recent publications on this subject were discussed in THE METALLURGIST, October, 1935, pp. 75–77.

depth of notch, in itself, did not affect the impact figure after it exceeded about one-quarter of the thickness of the test piece (Table). The effect of radius of notch in these and other experiments was variable, but increase of radius from 0·25 mm. to 0·5 mm. had a relatively small effect when the depth of notch was 2 mm. and over. Thus the two types of standard test piece are not likely to differ very much in sensitivity.

With a fixed total depth of specimen, *e.g.*, 10 mm., a large number of results show that the impact figure tends to be proportional to the depth below the notch in brittle materials and to the square of this depth in tough materials, usually lying between the two limits. For slight changes in depth of notch without any change of form, a reasonably close conversion can be made. To convert the values for a 3 mm. deep notch to those for a 2 mm. deep notch, the conversion factor will be between $\frac{8}{7}$ and $\left(\frac{8}{7}\right)^2$ that is, between 1·14 and 1·22. This approximate value (subject to a small correction, varying with the nature of the material, for change in radius of notch) may be expected to represent the ratio of energy absorbed by British standard and German sub-standard test pieces.

Three notch Izod test pieces of each form were cut from a number of nickel and nickel-chromium-molybdenum steel forgings and tested in the 120 foot-pounds Izod machine. The average impact figures of the British standard test piece are plotted against those of the German sub-standard in Fig. 4. The results are very scattered, ratios varying from 1·1 to 1·5 for nickel steels, and from 0·8 to 1·3 for nickel-chromium-molybdenum steels; but they appear to lie about straight lines giving average ratios of 1·2 and 1·1 respectively.

A further set of tests was carried out on a single nickel-chromium steel in different conditions of heat treatment. The results obtained under these conditions are much more regular, and show a tendency for the British standard figure to increase a little more rapidly than that of the German sub-standard. From this it seems that while the German sub-standard will discriminate well between tough and brittle steels, numerical differences in impact figure are just a little smaller than those obtained with the British standard test piece.

Aluminium in Modern Commercial Steels.

THE Edward de Mille Campbell Lecture of the American Society for Metals* was this year given by H. W. McQuaid, who chose as his subject "Aluminium in Modern Commercial Steels."

This is a subject which might no doubt be approached in different ways by different people, but the leading part played by the lecturer in the study and control of grain size in steels has largely determined the contents of the lecture.

Mr. McQuaid refers to his early experiments, with coarse and fine grain steels for case-hardening which preceded the introduction of the McQuaid-Ehn test. This test consists of carburising the steel to a depth of about 0·05in. in a solid carburising mixture at 1700 deg. Fah. (927 deg. Cent.), and subsequently cooling at a standard rate to develop a pearlitic structure. The grain size of the austenite is then outlined by a cementite network and the steel is classed as normal if the pearlite is lamellar and the network continuous, or abnormal if the pearlite is coarsely irregular and the grain boundaries consist of massive cementite and ferrite.

Normality and grain size are independent. Steels may be either coarse (normal or abnormal) or fine (normal or abnormal), according to the conditions of manufacture, but carbon steels are usually either coarse and normal or fine and abnormal.

In such steels the coarse grain, normal varieties respond better to normalising and to quenching, harden more deeply (though they are not so free from internal stress and distortion), and give a better finish on machining than the fine grain, abnormal varieties. On the other hand, they have a lower impact value after a given treatment than a similarly treated fine

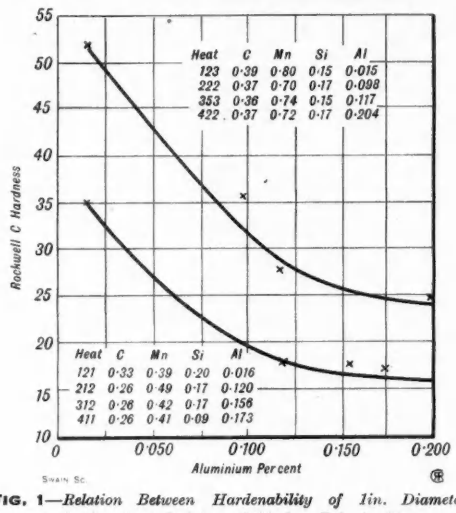


FIG. 1—*Relation Between Hardenability of 1in. Diameter Section, Quenched from 1700 deg. Fah. in Water, and Aluminium Content.*

grain steel. For this reason, and also to avoid distortion and cracking on quenching or grinding, fine grain steels are usually preferred in alloy grades intended for heat treatment. The tendency to grain growth is less and so a simple quenching operation may be adopted with these steels after a carburising treatment, although owing to their lesser hardening capacity soft spots increase in frequency in case-hardened fine grain, abnormal steel.

Mr. McQuaid goes on to record how, as early as 1922, aluminium was used for improving the toughness of carbon and alloy steels, and how its action was even then being closely correlated with its effect on grain size. He shows in a series of steels, selected because of their closely similar carbon, manganese, and silicon contents, that the hardness of the centre of a 1in. diameter section, quenched from 927 deg. Cent., is related to the aluminium content—Fig. 1—just as it is already known to be related to grain size. A set of curves, from which Figs. 2 to 4 are reproduced, sums up the conclusions relating to the comparative effect of manganese, silicon, and aluminium on depth hardening. Fig. 2 shows the effect of increase of manganese in increasing depth hardening through the

* Reported in *Iron Age*, October 10th and 17th, 1935.

thickness of a 1in. diameter bar quenched in water from 927 deg. Cent. The effect of increase in carbon in the high manganese material is shown by the top

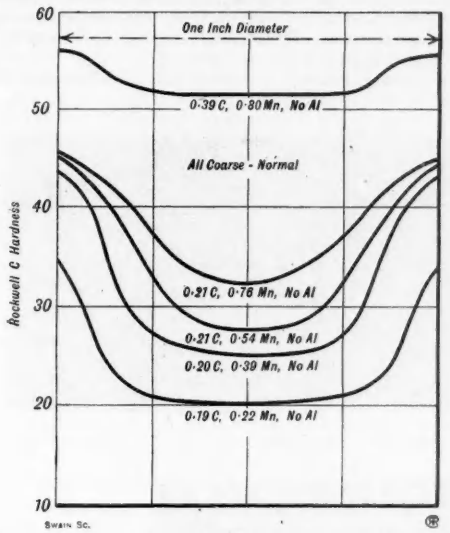


FIG. 2—*Effect of Increasing Manganese on 0·20 per cent. Carbon Steel without Aluminium Addition when Quenched in 1in. Section in Water from 1700 deg. Fah.*

line of the diagram. The effect of aluminium in reducing the grain size and simultaneously reducing the hardness at the centre of the 1in. diameter bar

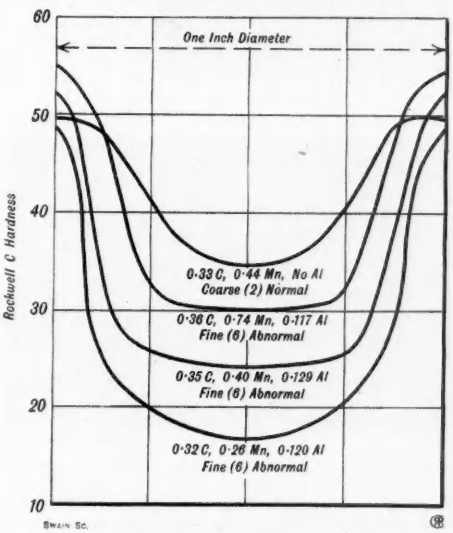


FIG. 3—*Effect of Increasing Manganese on a Medium Carbon Steel Containing Relatively Heavy Additions of Aluminium, when Quenched in 1in. Diameter Section in Water, from 1700 deg. Fah.*

is illustrated in Fig. 3 for steels of different manganese content. A similar effect of increasing aluminium is shown in Fig. 4.

It appears in practice that aluminium has but little influence in establishing fine grain characteristics until a definite minimum is added, and then the fine grain effect is obtained very rapidly. Hence, although coarse and fine grain specifications are relatively easy to meet, it is much more difficult, if not commercially impossible, to produce an intermediate grain size with regularity.

The lecture contains some speculations on the mode of action of aluminium in reducing grain size. It has been suggested, for example, by Bain in the Campbell Lecture for 1932, that sub-microscopic particles of alumina (or other insoluble oxides) obstruct grain growth and so produce fine grain steels with all the accompanying characteristics of small grain size. There are a number of observations, emphasising the

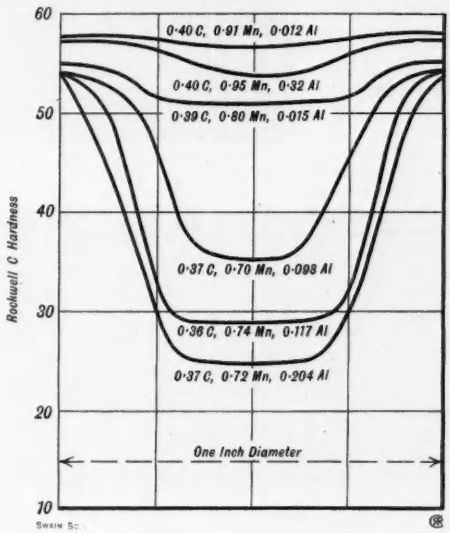


FIG. 4—*Effect of Increasing Aluminium Addition on Hardenability of Medium Carbon, Manganese Steel Quenched in 1in. Section from 1700 deg. Fah. in Water.*

importance of the time factor in the action of aluminium, which support this idea. For example, the effect of aluminium is diminished by holding a fine grain basic open-hearth steel in the ladle for a relatively short time. This suggests that the diminution of the effect is due to the coalescence and rising of particles of alumina.

Mr. McQuaid, while not denying that a dispersion of fine insoluble particles would inhibit grain growth, asks a number of questions, reflecting the result of actual practice, which imply that an excess of aluminium is required, over and above that necessary to form oxide with all the oxygen present, and that the excess aluminium is itself responsible for the fine grain, possibly indirectly through its influence on carbide solubility and on the coalescence of the carbide precipitated from austenite on cooling. The amount of aluminium which must be added in order to obtain fine grain characteristics depends on the degree of deoxidation of the bath before the addition is made ; thus the prior addition of manganese and particularly of silicon tends to reduce the amount of aluminium necessary.

In the same numbers of *Iron Age* there appears an

article on "Grain Size and its Influence on Steel Wire Manufacturing," by B. L. McCarthy. The behaviour of high carbon steel during wire drawing, considered in relation to its inherent grain size, leads to the conclusion that in the manufacture of wire in which high-tensile strength must be combined with good ductility, coarse grain steel, as recognised by the McQuaid-Ehn test, should be employed. The use of a coarse grain steel helps to promote a coarse grain patented structure, since the patenting temperature is above the grain-coarsening temperature. Fine grain steels are not desirable, because the patenting temperature is either below the grain-coarsening temperature or within the range of temperature in which non-uniformity of grain size develops, the result being the formation of fine grains with a tendency towards the formation of free ferrite and coarse pearlite. Mr. McCarthy points out that the McQuaid-Ehn grain size test may be employed to assist the open-hearth plant in planning the charge and working the heats. A practice which produces a wide variation in grain size in a certain type of steel indicates a poor operation. By using this test as a guide to quality, steel to meet the needs of the wire mill can be regularly produced.

Reference is made to this article here because Mr. McCarthy also refers to the effect of aluminium on grain size, and gives interesting examples of its effect in the structures shown in Figs. 5 to 7.

Coarse Grain Steel Carburised at 1600 deg. Fah. (on left), 1700 deg. (middle) and 1800 deg. (right) for 8 hr. The Grain sizes are No. 4, No. 3 and No. 1 from left to right respectively.

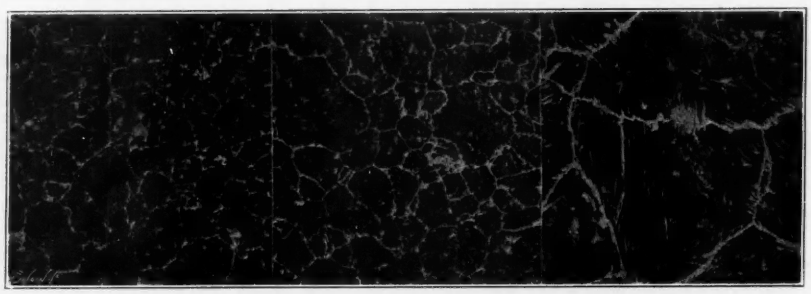
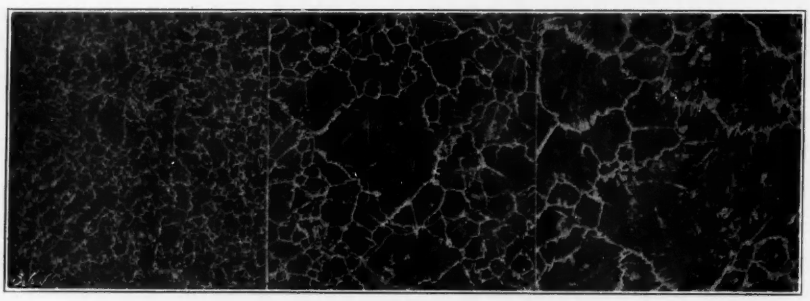


FIG. 5



Steel with 6·5 oz. of Aluminium per Ton Carburised for 8 hr. at 1700 deg. (on left), 1800 deg. (middle) and 1900 deg. Fah. (right). Grain sizes are No. 6, No. 4, and No. 2, from left to right respectively.

FIG. 6

Steel with 13 oz. of Aluminium per Ton, Carburised for 8 hr. at 1700 deg. (on left), 1800 deg. (middle) and 1900 deg. (right). Grain sizes are No. 7, No. 6 and No. 5, from left to right respectively.

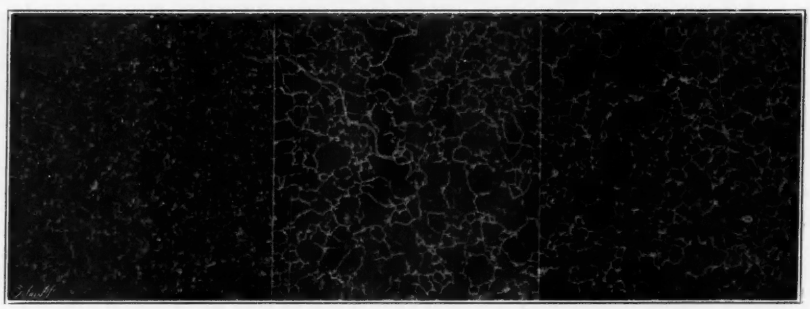


FIG. 7

The use of aluminium has involved the sacrifice of many old prejudices; in fact, it is stated by Mr. McQuaid that the secrecy which surrounded its use in the early years of grain size control by this method is to be accounted for by the publicity which had previously (*i.e.*, 1917 to 1920) been given to the disastrous effects of visible alumina inclusions on the

soundness, machinability, and other properties of good steel. There was also much opposition to, and even scorn of grain size specifications among prominent metallurgists. It was in America that the value of grain size specifications was first recognised, and there, according to Mr. McQuaid, such specifications have become so common as to be regarded as a natural method of governing the purchaser's requirements in the matter of machinability, absence of distortion on quenching, response to heat treatment, impact value, &c.

Although Mr. McQuaid implies that European steel producers have been behindhand in following up these developments, it is, nevertheless, a fact that British steel makers have long recognised the importance of grain size control for certain products, and that in this country steel can, when desired, be supplied to a grain size specification.

It could hardly be maintained that the Campbell Lecture for 1935 is written with the lucidity and conciseness of expression which have characterised some previous Campbell Lectures. Nevertheless, it admirably serves the purpose of bringing together much detailed information about the effect of aluminium additions, besides forming a valuable record, especially when read in conjunction with the Grain Size Symposium of 1934, of the important part which the author has played in this development.

Beryllium.

ISOLATED within a year of each other just over one hundred years ago, the two light metals, aluminium and beryllium, present interesting contrasts in regard to their present positions in the industrial field. With an annual production of over 280,000 tons, aluminium has shown a remarkable development, more particularly in the last twenty years, since a commercial process of extraction was introduced fifty years ago. On the other hand, it is only comparatively recently that, largely through the work of Stock and Gold, and Siemens and Halske A.G., beryllium has become a commercial product. Although the output of beryllium is at present limited to only a few tons per annum, and the price is very high, the history of aluminium gives rise to interesting speculations as to whether the next decade or so will see comparable developments in the production and use of beryllium. In a paper by Monsieur Robert Gadeau, presented to the Congrès International des Mines, de la Métallurgie et de la Géologie Appliquée, held in Paris in October, and a recent article in *Metall und Erz*,[1] interesting surveys are given of the present position of beryllium in industry, together with an outline of the methods of production, properties, and applications.

Production of the pure metal is at present in the hands of the Heraeus Vacuum Schmelze A.G. in Germany, the Beryllium Development Corporation in the U.S.A., and La Compagnie d'Alais, Froges et Camargue in France, the price per pound, according to latest figures, being approximately £13, £29, and £9 respectively (at par rates of exchange). Several factors contribute to the high cost. Thus, the principal ore, beryl, a complex silicate of beryllium and aluminium ($3BeO, Al_2O_3, 6SiO_2$) is widely disseminated, as in France, Madagascar, Canada, the United States, South America, and Russia, and its exploitation has not yet been placed on an economical commercial basis. Moreover, from this expensive raw material the yield of beryllium is barely 3 per cent., obtained only after a complicated preliminary chemical treatment and delicate electrolytic extraction process.

The chemically stable ore is first "opened out" by heating with sodium fluosilicate at 650–700 deg. Cent., giving a soluble double fluoride of sodium and beryllium, which, however, is itself a very stable compound and direct electrolysis of this has not been found practicable. After considerable research, methods have now been discovered for converting this compound to beryllium chloride, fluoride, or oxyfluoride, from which electrolytic extraction of the beryllium is possible.

In Germany the oxyfluoride ($5BeF_2\ 2BeO$), mixed with barium fluoride (BaF_2) to increase fluidity and reduce volatilisation losses, is electrolysed at 1400 deg. Cent.—*i.e.*, *above* the melting point of beryllium—in a graphite crucible which acts as anode. Beryllium, 98–99 per cent. pure, is deposited on a central, water-cooled iron cathode, which is gradually raised during the operation, giving a "carrot" of solid metal. Special auxiliary plant is necessary to recover the volatilisation losses, amounting to as much as 50 per cent.

The American process is somewhat different. A mixture of beryllium chloride and sodium chloride is electrolysed in a hydrogen atmosphere at 730–820 deg. Cent.—*i.e.*, *below* the melting point of beryllium. Particles of pure beryllium separate from the molten salt on to the sides of the iron-chromium or nickel-chromium crucible, acting as cathode, the anode consisting of graphite. The particles of pure metal are subsequently remelted.

Amongst the more interesting properties of the metal may be noted the very low density of 1·84, lying between that of aluminium (2·7) and magnesium (1·74), complete absence of ductility at atmospheric temperatures,* and a modulus of elasticity of 19,000 tons per square inch, and heat of fusion greater than for any other metal. It is readily volatilised above its melting point of 1278 deg. Cent., and, whilst inert to hydrogen, has a great affinity for oxygen, especially at raised temperatures, although at atmospheric temperatures the thin film of oxide forms a protective layer as in the case of aluminium. A permeability to X-rays seventeen times greater than that of aluminium has led to the only known application of the metal in the pure state, viz., as windows of X-ray tubes.

The chief application of beryllium in the metal industry lies in the production of alloys of low beryllium content, which are capable of being heat-treated to give outstanding mechanical and physical properties.

The lightness and high elasticity of the metal raised hopes of the development of strong light alloys for aircraft, &c., but so far the results of researches have been disappointing. Direct alloying with magnesium presents very serious difficulties, owing to the volatilisation of the magnesium at 1120 deg. Cent. Moreover, by indirect methods of preparation, Kroll has shown that the two metals separate out completely in the solid state. The occurrence of serious oxidation and gas absorption renders direct alloying of beryllium and aluminium impossible, although satisfactory "king" alloys can be prepared by adding aluminium fluoride to the electrolytic extraction bath. In any event, the effects of beryl-

* Sloman suggests that this is due to a Be/BeO eutectic surrounding the metal grains, and that pure Be is likely to resemble pure iron in mechanical properties (*J.*, Inst. Met., Vol. 49, page 365).

lium on aluminium appear to be without interest; the solid solubility of beryllium in aluminium is only 0·075 per cent. at 639 deg. Cent., falling to less than 0·013 per cent. at atmospheric temperatures. A eutectic is formed at 644 deg. Cent. with 1·4 per cent. beryllium.

So far, it is only in combination with the heavier metals that beryllium has given useful alloys. With nickel, the solid solubility decreases with temperature from 2·8 per cent. at 1155 deg. Cent., and with 2·0 to 2·5 per cent. beryllium the alloys can be age-hardened up to the very high figure of 600 Brinell.

Quite recently non-magnetic alloys of the "Nivarox" type, containing iron, nickel, molybdenum, and beryllium have been introduced in Germany[2] for use in timepieces. With 1 per cent. beryllium, age-hardening is induced by quenching from high temperatures and reheating. The high elastic limit and modulus of elasticity of hair springs made from these alloys allow a higher amplitude of the balance wheels and consequent improved accuracy of running. Such springs are sufficiently hard and strong to withstand the severest shocks without deformation, and with negligible temperature coefficient of elasticity, they are greatly superior to Elinvar springs. A similar type of alloy, called "Contracid Beryllium," containing nickel, chromium, molybdenum, and beryllium, is being used for main springs. It can be heat-treated to give thermo-elastic properties similar to those of hardened steel, whilst remaining non-magnetic and highly resistant to corrosion, and the springs have a uniform torque during almost the whole of the unwinding period. The alloy can also be used satisfactorily for certain moving parts of timepieces, which can be shaped in the soft, quenched condition, and hardened without tarnishing on reheating. Kroll has suggested the use of low-carbon steels, containing about 1·5 per cent. beryllium, and having high remanence, to replace silicon magnet steels.

Sloman[3] has discovered that additions of the order of 0·4 per cent. beryllium to standard silver containing 7·5 per cent. copper effect a considerable increase in resistance to tarnishing, particularly in moist sulphurous atmospheres. The alloy can also be improved slightly by age-hardening.

The most firmly established alloy, having the widest application, is that consisting of copper with 2·0 to 2·5 per cent. beryllium and known as "beryllium-bronze" or, more correctly, "beryllium-copper." Preparation by direct mixing is not possible owing to oxidation of the beryllium. "King" alloys can be prepared in the electrolytic extraction bath by adding copper pellets to form first copper fluoride, which is then decomposed with the beryllium salt, or by using a cathode of molten copper at the bottom of the crucible and an anode of carbon. The work of Masing and Dahl, Borchers and others has shown the solid solubility of beryllium in copper to vary from about 2·7 per cent. at 860 deg. Cent. to less than 1·0 per cent. at atmospheric temperatures—see figure—and for this reason the properties of the alloys can be varied considerably by heat treatment. Quenched after heating in a hydrogen atmosphere at 800 deg. Cent., the alloys are very soft (about 100

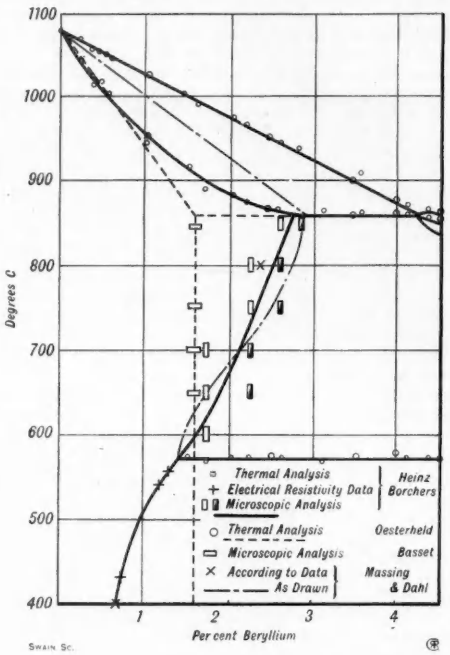


TABLE.—*Mechanical Properties of Beryllium Alloys.*

Alloy.	Condition.	Yield point, tons per sq. in.	Maximum stress, tons per sq. in.	E, tons per sq. in.	Elongation, per cent.	Brinell hardness.	Charpy impact figure, ft.-lb. absorbed.
Beryllium-copper, 2·0 to 2·5 per cent. Be	Quenched	13·8	31·2	8,030	45·0	110	59·7
	Quenched and heat treated	59·8	78·1	8,430	6·3	340	7·3
	Quenched and work-hardened	46·9	52·6	7,680	4·3	220	16·7
	Quenched and work-hardened and heat-treated	61·6	86·1	8,220	2·0	365	6·1
Beryllium-nickel, 1·9 per cent. Be	Quenched	22·8	51·5	11,120	42·9	140	—
	Quenched and heat-treated	48·6	78·7	11,630	25·3	365	—
	Quenched and work-hardened	93·8	98·1	11,430	1·5	350	—
	Quenched and work-hardened and heat-treated	95·3	116·0	12,080	8·3	460	—
Contracid Beryllium	Quenched	26·5	55·9	9,850	29·5	195	—
	Quenched and heat-treated	31·5	59·3	10,230	22·8	320	—
	Quenched and work-hardened	89·0	95·3	10,730	2·0	320	—
	Quenched and work-hardened and heat-treated	95·3	117·8	11,760	6·0	430	—

Brinell) and can be produced in many worked forms, such as plates, thin sheet, strips, bars, wire, die-pressed forgings, and are also easily machinable. The microstructure of the quenched alloy consists of the α solid solution together with a small amount of the β constituent. The American Brass Company add 0·25 to 0·5 per cent. nickel to reduce the large grain size, which normally results from the first solution treatment. On subsequently reheating above 175 deg. Cent. considerable hardening occurs with a maximum effect at 300–325 deg. Cent. The actual temperature and time of reheating are varied according to the properties required. The structure is completely altered by the appearance of a lattice-like pattern in the α grains, due to a colloidal or sub-microscopic precipitation, probably γ, on definite crystallographic planes; the highest magnification fails to reveal particles of the precipitated phase.

The outstanding mechanical properties of beryllium-copper are indicated in the table, where the corresponding figures for beryllium-nickel and "Contracid Beryllium" are given for comparison.

The other useful properties include a high fatigue limit of 15–18 tons per square inch, compared with 11–12 tons per square inch for phosphor-bronze and very good resistance to wear. In comparison with steel, beryllium-copper is notable for its conductivity, resistance to corrosion, machinability, and non-magnetic and non-sparking properties.

According to a very interesting booklet issued by the American Brass Company, manufacturers of Anaconda beryllium-copper, some of the applications of the alloy include clips, switch blades, vibrator arms, contact brushes, motor brush springs; non-sparking hand tools; bolts, screws, precision bearings and bushes and ball cages; watch bearings to replace jewels; firing pins; welding electrodes; springs of all types; diaphragms and woven wire cloth.

The electrical conductivity of heat-treated 2·3 per cent. beryllium-copper is only about 25 to 30 per cent. of that of copper, but it has been found[4] that, with the addition of cobalt (one-twentieth the price of beryllium), not only can similar mechanical properties be obtained by heat treatment, with greatly reduced beryllium content, but the conductivity is increased to 50 per cent. of that of copper. The standard alloy which has been developed by the G.E.C. is only one-third the price of beryllium-copper. Moreover, the precipitation temperature is raised to about 500 deg. Cent., so that the alloys are more stable at raised temperatures. The alloys can be produced in any form in which brass is used and the unique combination of strength and conductivity renders them particularly valuable for welding electrodes for heavy current and high pressures, soldering iron tips and jaws, springs with mechanical and electrical loads and pole shades.

Thus already in the industrial field beryllium has proved to be an interesting and valuable new metal, and with further investigations leading to lowering of the costs of production and the development of a wider range of useful alloys, it is likely to become of increasing importance in the metallurgical and engineering industries.

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Rarer Elements in Metallurgy.

THAT no fewer than twelve papers in the last volume of the "Transactions" of the American Electro-Chemical Society are devoted to the electro-chemistry of the rarer elements, in some instances with extended references to their metallurgical applications, is at least an indication of the increasing interest which is being taken in materials of this kind. While some of the rarer elements are at present very costly, it is, nevertheless, extremely desirable that their properties and possible industrial applications and uses should be ascertained, for whilst they may be expensive to-day and outside the possibility of any but the most limited and very special sort of application, it may well be, in some cases at least, that the discovery of more abundant sources of supply and improved methods of extraction will render them relatively cheap. There have been instances of this in the past and there is no reason for supposing that this experience may not be repeated. A number of metals which a few years ago were regarded as rare are now so generally used that they are looked upon as metallurgical commonplaces. Such elements as cobalt, molybdenum, tungsten, and silicon are good examples, while three metals, namely, nickel, aluminium, and mangesium have long passed beyond the stage of being special alloying elements, but serve each as the basis of a whole group of very important alloys. In metallurgical research a vast unexplored field exists in so far as the rarer elements are concerned. It is no argument against the prosecution of work in this direction to insist that our knowledge of well-known alloys is still very imperfect, or that many of the rarer elements are indeed extremely rare and never likely to be anything else. Gaps in our knowledge of the properties of existing alloys are continually being filled, and the results which have been obtained in research in recent years have, in fact, led to the development of many new industrially useful alloys. Whilst there may yet be surprises in store with regard to the alloying possibilities of the more commonly used metals, yet it is only reasonable to look for more rapid progress in newer fields of investigation and an attitude of hopeful expectancy towards the rarer or newer metals is very understandable. In recent years the basis metals used for alloy manufacture, such as, for example, iron, copper, lead, zinc, aluminium, tin, and nickel, have been produced in much higher degrees of purity than they were formerly, and in many instances the alloys now produced are not quite the same either in their properties or in their response to fabricating treatments as they were some years ago, a difference which is probably attributable to the difference in purity of the alloying elements. This perhaps to some extent accounts for the more systematic work which has in recent years been carried out on the effect of varying amounts of different elements, often the commonly occurring impurities or the usual alloying additions, on the properties of metals and alloys. It is but a logical step to consider next the effect of rarer substances. The possibilities of the use of comparatively rare elements appear to have been explored more in connection with steel production than in non-ferrous metallurgy. Tungsten, nickel, chromium, molybdenum, and cobalt are now quite commonly used in the preparation of alloy steels, and rarer elements such as cerium, vanadium, titanium, lithium, calcium, and columbium are now being used to some extent. Some of the results are sufficiently promising to suggest

that there no doubt will be further developments in these directions. Many of them have been tried as deoxidants, and in this connection some of them appear to have virtues which extend beyond effective deoxidation only, for they remove gases from the melt and refine the grain. In some instances they are added not only as cleansers and for their scavenging action, but because of their beneficial effects on the mechanical and corrosion-resisting properties of the alloys. In non-ferrous metallurgy an interesting new development involving the use of a material which has hitherto been a stranger to ordinary metallurgy is the production of tellurium-lead alloys. Beryllium, a relatively rare metal, is being used commercially to some extent in the production of several alloys. Such substances as calcium, lithium, barium, and titanium are being used to increasing extents. The first three are used in the manufacture of lead-base bearing metals. Calcium is a very effective agent for de-bismuthising lead, and it is used as an alloying addition to lead for raising its fatigue resistance. Calcium and lithium are used as deoxidants for copper, while cerium and thorium are quite well known in some metallurgical applications. Tantalum is another uncommon element which is being used to some extent. Many other instances could be cited where the rarer elements are being used commercially. The development of electrical industries and wireless transmission has resulted in commercial applications being found for several of the rarer metals, and some, notably the precious metals, are being used in plating. With the further development of age-hardenable alloys, an increased use of some of these rarer elements can reasonably be anticipated, and known examples certainly suggest that further uses will be found for them in the production of alloys for special purposes where properties are required which are not possessed by the more ordinary or usual alloys.

It will be remembered that some years ago there was in existence a Committee of the Department of Scientific and Industrial Research known as the Minor Metals Committee, which was formed in 1922 as a result of representations made by the Imperial Mineral Resources Bureau. This Committee directed some useful work on cadmium, and as a result accumulated data which proved to be of great value in the subsequent industrial development of alloys of that metal. It supervised all the work on metallic beryllium which was done in this country, and was responsible for some new applications of titanium compounds. Other elements came under review, but the Committee was disbanded in 1931, largely, it is believed, as a measure of economy. Metallurgical research on the rare metals and their alloys is still, however, a matter of national importance, which deserves to be encouraged. The possibility of practical application of much pioneer work, which may ultimately be very fruitful, is often too remote to secure for it a high degree of priority in the programme of an industrial research organisation.

Refined Aluminium.

ALUMINIUM produced by the Héroult process usually contains at most 99·6 per cent. of the metal, a percentage which by the use of specially purified alumina and pure carbon anodes may be raised to 99·85. Many metals, for example, zinc, copper, tin, and iron, can be purified to a higher degree by electrolysis from aqueous solutions, but this method is, of course, not applicable to aluminium, which in these circumstances would decompose water giving a precipitate of aluminium hydroxide. Hence for a considerable time efforts have been directed towards the attainment of higher purity in aluminium by the electrolysis of fused salts.

This matter was discussed by Monsieur Robert Gadeau at the recent International Congress at Paris, in a paper entitled "Manufacture, Properties, and Uses of Refined Aluminium." He first referred to the early suggestion of Betts (1905) for a three-liquid-layer cell, the lower layer or anode being an alloy of aluminium, the upper layer or cathode the refined aluminium, and the middle layer the electrolyte composed of salts of aluminium to which some heavier salts had been added to produce a density intermediate between that of the liquid aluminium cathode and the liquid alloy anode.

In a cell thus formed the current would transfer aluminium from the lower to the upper layer. Metals more electro-negative than aluminium (iron, silicon, copper, manganese, &c.), are not dissolved at the anode, and remain in the alloy at the bottom, while those more electro-positive (magnesium, calcium lithium, &c.), are dissolved at the anode and pass into the bath, but remain there, and are not liberated at the cathode. Although this principle of refining is simple, its industrial application presents numerous difficulties, and it was a great many years before the idea took practical form. Hoopes first obtained aluminium by this process.* The base of the cell consists of a carbon lining on which rests the molten anodic alloy containing 33 per cent. of copper. The electrolyte employed is a mixture of cryolite and barium fluoride containing alumina in solution, and having the composition :—

	Per cent.
Aluminium fluoride (AlF_3)	25 to 30
Sodium fluoride (NaF)	25 to 30
Barium fluoride (BaF_2)	30 to 38
Alumina (Al_2O_3)	0·5 to 3·0

This bath has a high melting point, and must therefore be worked at 950 deg. to 1000 deg. Cent. At this temperature the only refractory material resistant to a fluoride bath is carbon, but this will not serve as the lining of the sides of the cell since the upper and lower metallic layers must be insulated electrically. For this reason the Hoopes cell is provided with insulated water-cooled sides which become thickly encrusted with the solid electrolyte. By this process it has been possible to prepare refined aluminium having a purity of 99·90 per cent., and small quantities have attained a purity of 99·99 per cent.

One of the principal difficulties of the process arises from the slight solubility of alumina in the bath, so that any cooling, either accidental or due to the water-cooled sides, causes the formation of crystalline deposits of alumina, which do not readily redissolve. The crust which forms on the water-cooled sides has not the same composition as the bath, and any change in its thickness results in a change in composition of the liquid. Moreover, variations in temperature may cause solid portions of the crust to fall off and come to rest on the surface of the anodic alloy. The current density is thereby locally disturbed and copper and other impurities pass into the electrolyte with the ultimate effect of contaminat-

* It is claimed that Hoopes' original proposition ante-dated the patents granted to Betts (see "The Aluminium Industry," by Edwards, Frary and Jeffries, 1930, page 321).

ing the cathode. Thus for many years the regular functioning of the cell and the purity of the product have been detrimentally affected by conditions which arise out of the necessity for water cooling.

Monsieur Gadeau then describes the process adopted by the Compagnie d'Alais, Froges et Camargue, with which he is connected.

The principle of the three liquid layers is retained. The bath consists of :—

	Per cent.
Aluminium fluoride	23
Sodium fluoride	17
Barium chloride	60

The molecular ratio of AlF_3 to NaF is as 1 to 1·5, instead of 1 to 3 as in cryolite. This bath melts at so low a temperature that it can be operated between 700 deg. and 750 deg. Cent. The difference in density of the liquid layers (viz., molten aluminium 2·3, electrolyte 2·7, molten 33 per cent., copper-aluminium alloy 3·0), is sufficient to ensure that there will be no mixing. The A.F.C. cell is shown in Fig. 1. The sides are lined with magnesia bricks. These are not

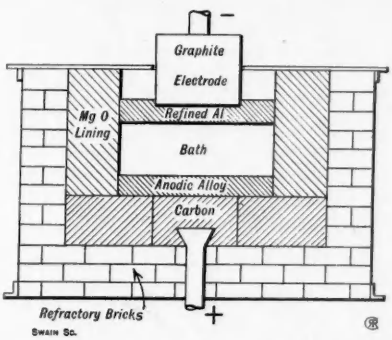

FIG. 1—*Diagram of Cell Used by the Compagnie d'Alais, Froges et Camargue.*

attacked by the bath, since barium chloride does not attack magnesia, but, in fact, protects it from the attack of fluorides at the working temperatures.

As there is no cold region, all the electrolyte remains molten, its composition does not vary owing to partial solidification, and it does not form any solid deposit on the surface of the anodic alloy.

The electrolytic decomposition potentials of the constituents of the bath are $BaCl_2$, 4·4 volts ; AlF_3, 4·8 volts ; and NaF, 5·9 volts. It is probable that barium is first formed at the cathode, and then reacts with aluminium fluoride to give barium fluoride and metallic aluminium. At the anode chlorine reacts with aluminium without attacking the impurities, and forms $AlCl_3$, which then becomes the active agent in electrolysis, since its decomposition potential is only 2·33 volts. Since aluminium chloride is volatile, a certain amount escapes into the atmosphere, and, in order to replace it more barium chloride is electrolysed. Thus the bath is slowly improvised in chlorine and aluminium, but tends to become richer in BaF_2, hence the aluminium sodium fluoride part of the bath tends, little by little, to approach the composition of cryolite $AlF_3 . 3 NaF$. If the bath is allowed to run until this proportion is reached, the cathodic aluminium is found to contain 3 per cent. of barium. The barium is unable to replace the aluminium in a comparatively stable compound, like cryolite. If a little aluminium fluoride is added to the bath, however, the barium in the refined aluminium re-enters the bath, and is not again separated so long as the correct composition of the fused salts is maintained.

The product usually contains more than 99·99 per cent. aluminium (iron 0·005, silicon 0·004, copper 0·001 per cent.). Most of it is 99·995 per cent., and the purest so far made 99·9986 per cent. (iron 0·0002, silicon 0·0009, copper 0·0003 per cent.).

The pure metal is very soft. Gadeau gives the following results of tensile tests on 99·99 per cent. aluminium :—

	Tensile strength, tons per sq. in.	Elongation, per cent.
As cast	3·2	45
Worked and annealed for three hours at 350 deg. Cent.	3·5	55–60

It may be recalled that the mechanical properties

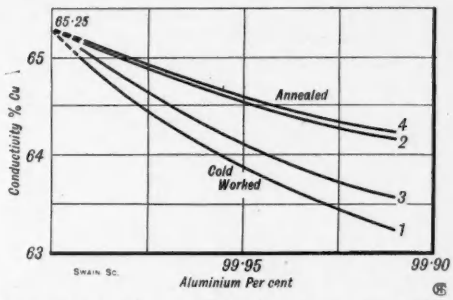

FIG. 2—*Conductivity of Refined Aluminium. 1 and 2, Cold Rolled to 12 mm. then Cold Drawn to 8·5 mm. 3 and 4, Hot Rolled to 12 mm. then Cold Drawn to 8·5 mm. 2 and 4 Annealed 3 hours at 350 deg. Cent.*

of aluminium of the highest degree of purity in relation to cold work and annealing were investigated by J. Calvet.[2] He found that aluminium recrystallised at lower and lower temperatures as the purity increased. At about the same time, J. J. Trillat and M. Paic[3] endeavoured to determine the recrystallising temperatures of specimens of cold rolled aluminium of various degrees of purity by an X-ray study of the materials, but obtained unduly high values. They, however, continued in collaboration with Calvet[4] experimenting on 99·9986 per cent. aluminium in the form of strip 1·08 mm. thick, rolled from an ingot 22·9 mm. thick, and were able to report as follows :—

Deg. Cent.	Distinct evidence of crystallisation. After	Recrystallisation complete. After
At 0	12 hours	—
25	1 hour	48 days
40	½ hour	48 hours
60	—	6 hours
100	—	1 min.

At temperatures above 150 deg. Cent. the recrystallisation of this material was almost instantaneous.

Gadeau[1] gives values for the electrical resistivity

[1] R. Gadeau, International Congress of Mining, Metallurgy, and Applied Geology, Paris, 1935 ; also *Rev. Aluminium*, 1935, 12, 2831 ; *Aluminium*, 1935, 17, 513 ; *Chim. et Ind.*, 1935, 34, 1021.

[2] J. Calvet, *Comptes Rendus*, 1935, 200, 66 ; THE METALLURGIST, April, 1935, page 26.

[3] J. J. Trillat and M. Paic, *Comptes Rendus*, 1935, 200, 1037.

[4] J. Calvet, J. J. Trillat, and M. Paic, *Comptes Rendus*, 1935, 201, 426.

of pure aluminium (Fig. 2), and results illustrating the effect of increasing purity on its resistance to chemical attack (Fig. 3). Aluminium of 98·5 per cent. purity becomes completely disintegrated in concentrated hydrochloric acid and in hydrochloric acid (sp. gr. 1·094) in half an hour; in hydrochloric acid (sp. gr. 1·017) in three hours, the loss of weight being 11 gr. per dm.^{2} On the other hand, refined aluminium (99·99 per cent.) lost only 0·06 gr. per dm.^{2} in sixteen hours in concentrated hydrochloric acid,

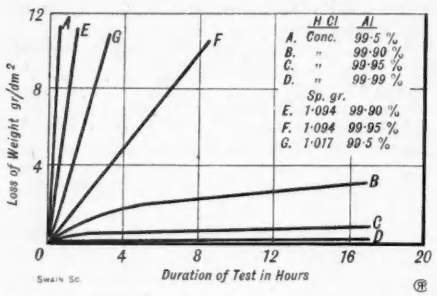

FIG. 3—*Chemical Attack by Hydrochloric Acid.*

and 0·044 gr. per dm.^{2} in fifty-two hours in the dilute acid.

The possible uses of refined aluminium are discussed. Its high resistance to corrosion and great pliability make it suitable, in the form of foil, for packing cheese and other foodstuffs. It has been used for electrical condensers. Another interesting development, as yet little studied, is its use in the preparation of alloys of high purity. Remarkable resistance to corrosion has already been observed in magnesium aluminium alloys (MG 7) prepared from the purest metals available.

Books and Publications.

Corrosion: Causes and Prevention. By F. N. SPELLER. London: McGraw-Hill Publishing Company, Ltd. 1935. Price 42s. net.

THE second edition of Dr. F. N. Speller's book on "Corrosion: Causes and Prevention," adheres to the general plan adopted in the first, in that the work refers mainly to ferrous metals and alloys, and is divided into two parts, dealing with the principles of corrosion and with its prevention in various detailed fields of application, respectively. The book has, however, been considerably enlarged, rewritten in numerous places and brought up to date. It is to be regarded as a notable contribution to the literature of corrosion, on which Dr. Speller is to be heartily congratulated.

In the first part of the work, Dr. Speller discusses the general principles of corrosion under the headings:—Theories of corrosion, influence of methods of manufacture and treatment, influence of factors internal to the metal (composition), influence of factors external to the metal (classification of corrosion), principles of corrosion testing, and relative corrosion of ferrous metals. Most, if not all, the statements made in this section will probably be accepted by other authorities on the subject, although there are naturally some minor points on which differences of opinion may occur. For example, the reviewer does not agree with the statement that a natural protective film forms on zinc as a result of atmospheric corrosion, since the experimental work of Vernon and of Hudson tends to show that the corrosion of zinc is practically proportional to the time, at least in appreciably polluted atmospheres.

The second part of the book is devoted to a discussion of preventive measures, and should make a strong appeal to the architect and the engineer. There are chapters on the prevention of corrosion in the atmosphere, underwater, underground, and in chemical industries, respectively, all full of sound practical advice, gained from the author's own experience and from other sources. Perhaps, however, the most noteworthy chapters in this section are those dealing with the prevention of corrosion in closed water systems, in high-pressure steam plants, and in steam and hot water heating systems, since, in view of his close association over a period of twenty years with investigations of the corrosion of tubes and pipes, Dr. Speller writes with particular authority on these subjects. He gives several examples illustrating the marked progress that has been made in recent years in reducing corrosion in these types of plant—a development in which he himself has played an important part. Indeed, the whole book gives a vivid impression of the progress effected within the last five or ten years in reducing the wastage from corrosion. This progress, as the author truly states, has been closely connected with the rapid development of stainless steels, which are playing an increasingly important part in chemical industry, and the introduction of low alloy structural steels containing small amounts of copper.

There are one or two subjects that one could have wished Dr. Speller to discuss more exhaustively, since his views and experience of them would have been of much value. For example, the subject of pickling, with all its ramifications, such as hydrogen absorption, embrittlement, and the use of inhibitors, is of much direct interest to the industry, and a more extensive statement of the present views and state of development in America would have been particularly welcome. Marine corrosion, as affecting shipping, is not extensively discussed, whilst corrosion fatigue, more particularly from the standpoint of the recent work of Gough and his associates, and the high-temperature oxidation of metals, including the effect of gases and vapours at the high temperatures and pressures used in chemical syntheses, are perhaps not treated so fully as their importance would warrant. There are also several omissions from the selected bibliography given at the end of the volume, in which, for example, the names of Bablik, Bardenheuer, Chaudron, Herzog, Palmaer, and Portevin will be sought for in vain, as will also any reference to the conferences on corrosion (*Korrosionstagungen*), which have been held annually in Germany since 1931.

These, however, are relatively minor points, and on each of the many subjects with which he deals, Dr. Speller's remarks will be found interesting, valuable, and instructive. Both by itself and as a basis for further study, this is an admirable book, which can be thoroughly recommended, not only to those engaged in corrosion research, but also to all who are responsible for the construction or maintenance of any type of steel structure.